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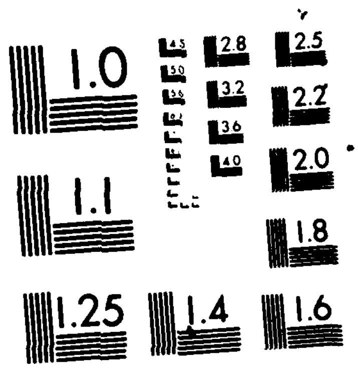

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A unique glove box system has been built which permits the melting and fabrication of halid and chalcohalide glasses under controlled atmospheres. Glass fibers have been prepared inside this system and their tensile strengths and viscoelastic properties measured in situ. Further the microstructures of glasses and ceramics prepared as well as their infrared transmission were investigated without the need to expose the samples to the external atmosphere. The system has been used to perform research for the Air Force Office of Scientific Research and to train students in the preparation of ultrapure glasses and ceramics.

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UNIVERSITY OF CALIFORNIA, LOS ANGELES
LOS ANGELES, CALIFORNIA

FINAL TECHNICAL REPORT

to

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

on Research Instrumentation Grant

PREPARATION PROPERTIES AND CHARACTERIZATION OF ULTRAPURE GLASSES AND CERAMICS

Grant No.: AFOSR-85-0121

Inclusive Dates: January 1, 1985 to January 31, 1986

Principal Investigator: Dr. John D. Mackenzie
Professor of Engineering and Applied Science

August, 1986

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ABSTRACT

An unique glove box system has been built which permits the melting and fabrication of halide and chalcohalide glasses under controlled atmospheres. Glass fibers have been prepared inside this system and their tensile strengths and viscoelastic properties measured in situ. Further the microstructures of glasses and ceramics prepared as well as their infrared transmission were investigated without the need to expose the samples to the external atmosphere. The system has been used to perform research for the Air Force Office of Scientific Research and to train students in the preparation of ultrapure glasses and ceramics.

I. Introduction

Prior to approximately 1960, glasses of ultrapurity were relatively unimportant. Subsequently, the development of waveguides created needs for ultrapure oxide glass fibers. The development of infrared transmitting glasses created needs for ultrapure chalcogenide glasses. Recently, it has been recognized that *Fluoride Glasses* are potentially optical materials of great importance because of their unique properties. Similar to the oxide and chalcogenide glasses, impurities can play a major role in dictating the properties of fluoride glasses. In particular, water, oxygen and transition metal ions are of importance. At present, there is little knowledge on the quantitative effects of these purities on optical, mechanical and chemical properties of the new fluoride glasses. In order to perform meaningful quantitative research such as the relationship between property and chemical composition of fluoride glasses, ultrapure samples must be available and the effects of impurities must be ascertained. Samples must be prepared in a highly purified atmosphere without water and/or oxygen. Graduate students must also be trained to process ultrapure glasses for research and development.

Up to 1985, no university laboratory in the U.S. had a truly outstanding capability of processing ultrapure fluoride glasses in controllable atmospheres, to characterize the samples without having to remove them from the inert atmosphere, to measure some important properties such as tensile strength in the same atmosphere and to fabricate ultrapure samples for evaluation by other laboratories. The need for a university laboratory with such capabilities for fluoride glasses, and other halide glasses, was obvious and urgent.

Another relatively new area of materials research which hold great promise is the preparation of glasses and ceramics by the so-called "Sol-Gel" method. Here, the control of the ambient atmosphere, especially the amount of water is of utmost importance to prepare pure glasses and ceramics. Again, the control of ambient atmosphere during the processing of the gels is very desirable. No extensive capability for these purposes was known to exist in U.S. universities in 1985.

A proposal was therefore submitted to the Department of Defense for funds to establish an unique facility at UCLA for the preparation and characterization of ultrapure halide glasses, other glasses and ceramics. Briefly, the facility would contain a system of interconnecting dry-boxes which would permit the preparation of halide glasses under controlled

atmospheres, fabrication of the glasses into monolithic samples or fibers and the measurement of some properties, all under the protective atmosphere.

The proposal was approved in the latter part of 1984 and funds were made available in January 1985. The total award was $225,000. This report describes the equipment purchased with the funds, the assembling of the facility and the uses made with our unique facility.

II. Description of the Facility

The facility consists of five connected glove boxes, one old one which was renovated and four new ones purchased under the present grant. A schematic drawing of the system is shown in Figure 1. Figures 1, 3 and 4 show the glove boxes in the directions AA, BB and CC of Figure 1. Closer views of boxes No. 2, 3, 5 and 4 are shown in Figures 5, 6, 7 and 8 respectively. The functions of these five glove boxes are described in Table 1.

In addition to the dry boxes, other equipments were purchased. These are described in Table 1. The equipments purchased under this grant, the vendors and the costs are shown in Table 2. All the equipment was delivered by September, 1985. However, modifications of the glove boxes had to be made and these were completed in March, 1986. The entire system was in operation in April, 1986.

III. Research Performed with the Facility

In addition to the Principal Investigator (Professor J. D. Mackenzie), fourteen members of the ceramics laboratory at UCLA have been using the equipment purchased to perform their research. Their names and the equipment used are shown in Table 3.

Since the glove box system was completed in March, 1986, the following research tasks have been performed *inside* the system:

1. Preparation of new halide and new chalcohalide glasses.

2. Measurement of tensile strengths of glass fibers.

3. Study of viscoelastic deformation of halide glass fibers.

4. Study of microstructure of halide and chalcohalide glasses by optical microscopy.

5. Study of infrared transmission of halide and chalcohalide glasses.

The other items of equipment shown in Table 2 were utilized for the study of halide glasses, chalcohalide glasses, sol-gel derived glasses and ceramics, polymer-oxide composites and glass-ceramics.

IV. Summary

An unique glove box system and supporting characterization equipment were obtained at UCLA and put into successful operation. Many new glasses have already been prepared, characterized and their properties measured. This facility will enable the Ceramics Laboratory at UCLA to investigate the effects of impurities and ambient atmospheres on halides, chalcogenide, chalcohalide and oxide glasses derived from gels.

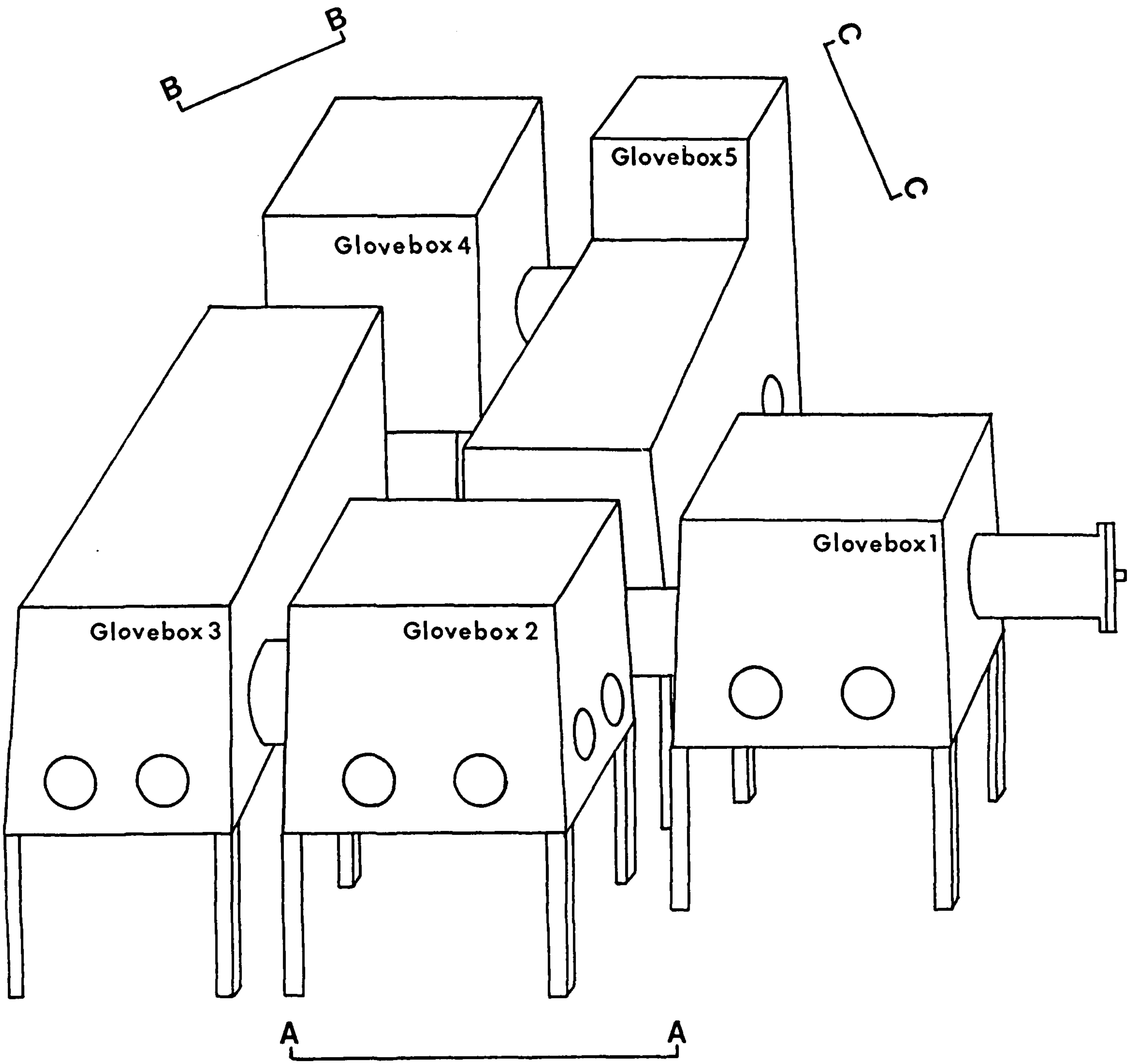

Figure 1. Glove box system.

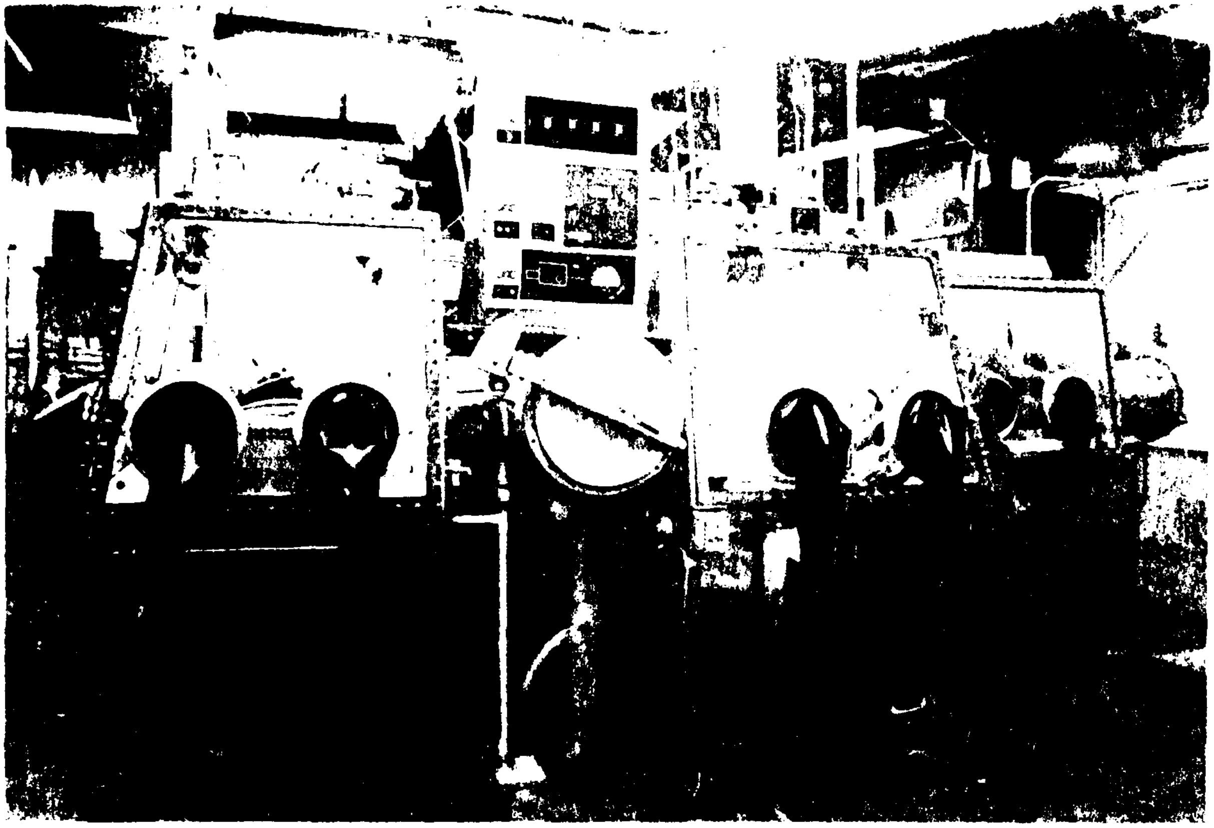

Figure 2. View of Glove Boxes in AA direction.

Figure 3. View of Glove Boxes in BB direction.

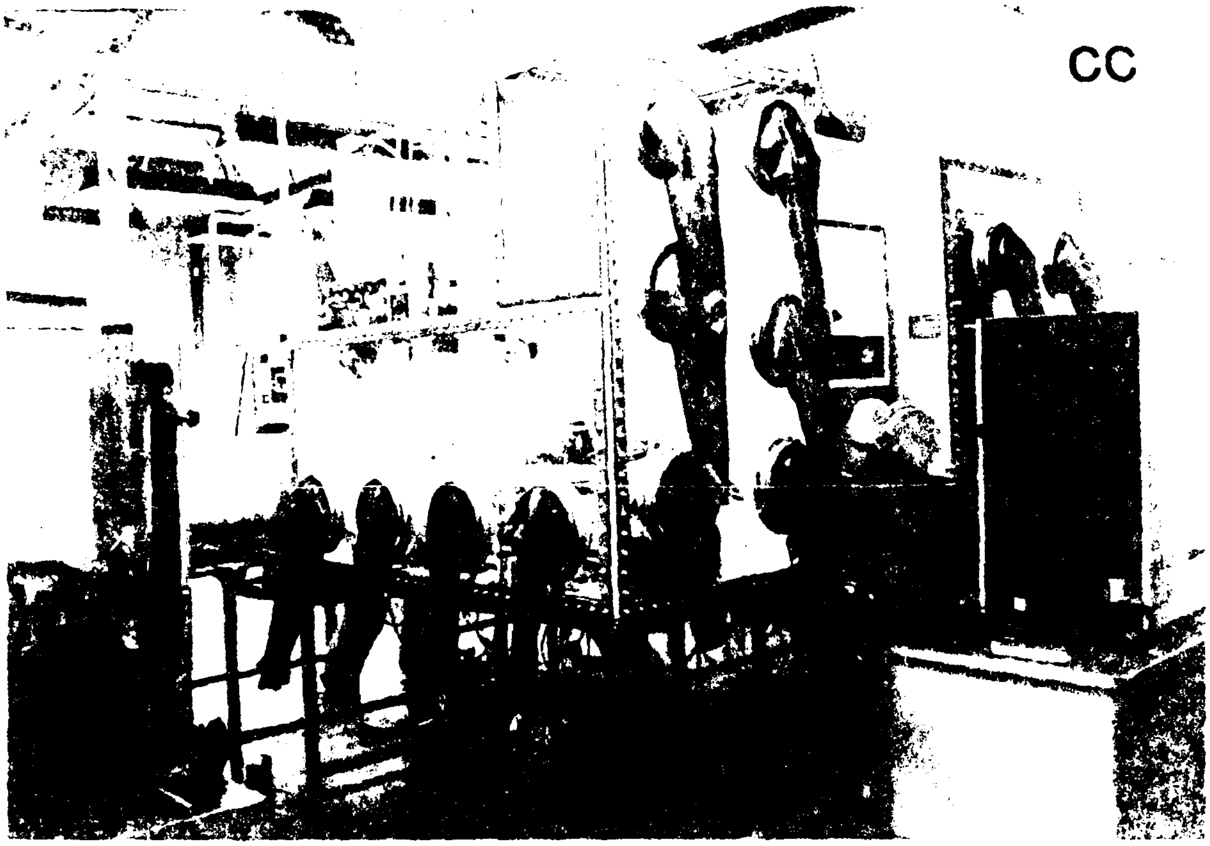

Figure 4. View of Glove Boxes in CC direction.

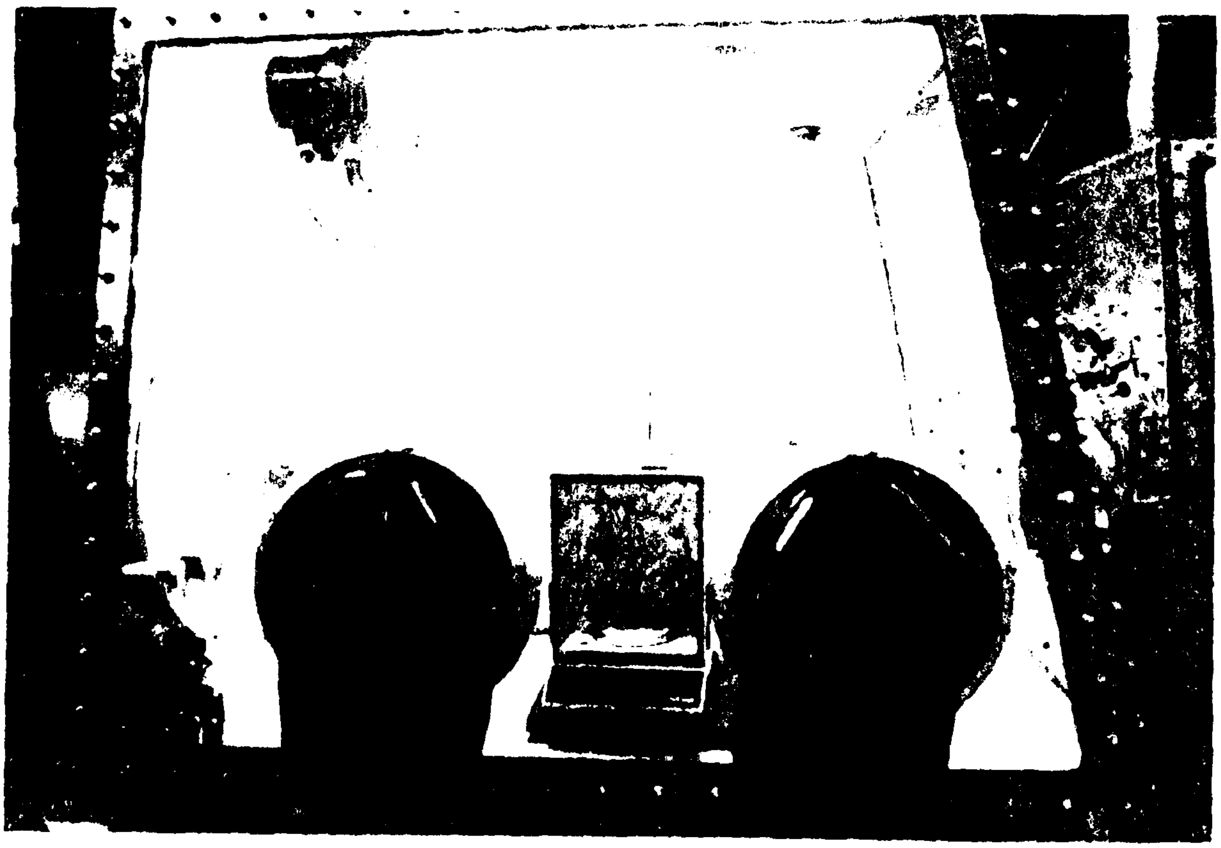
Figure 5. Close view of Box No. 2.

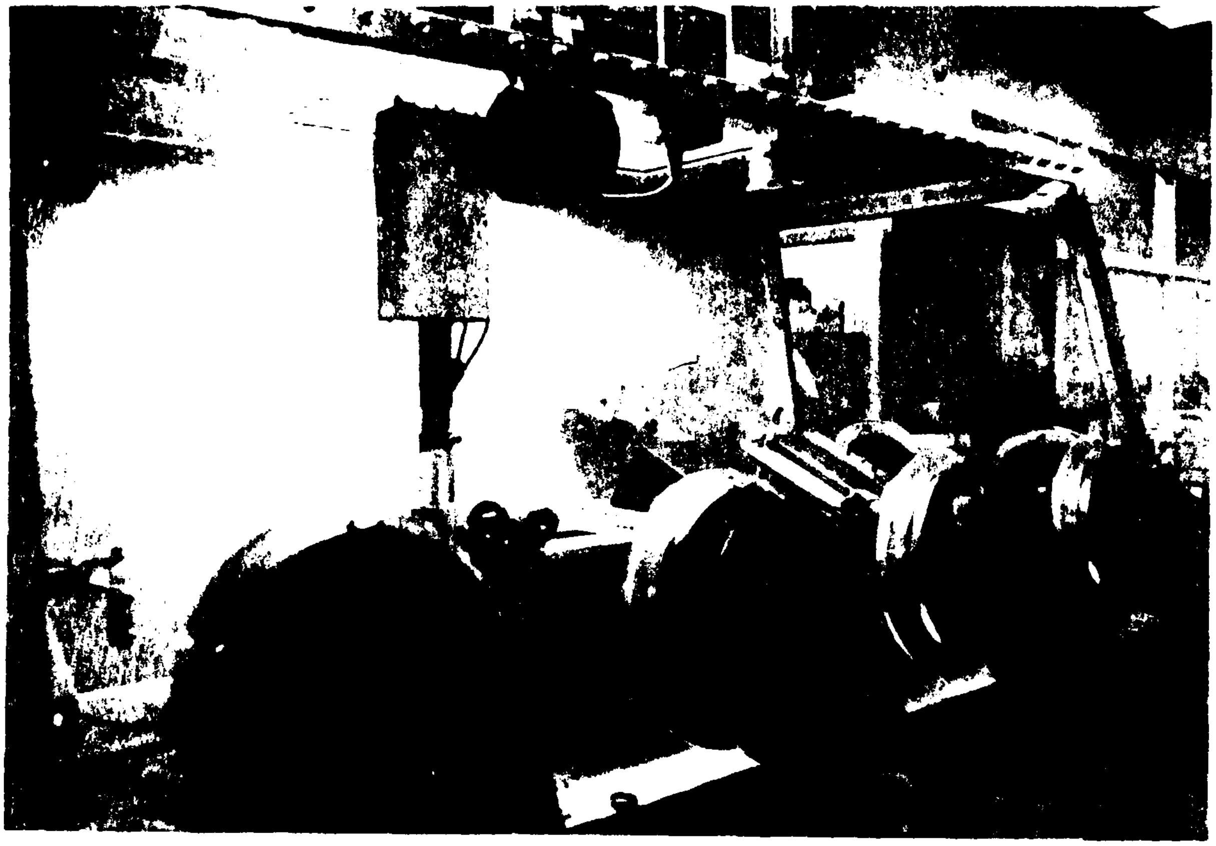

Figure 6. Close view of Box No. 3

Figure 7. Close view of Box No. 5

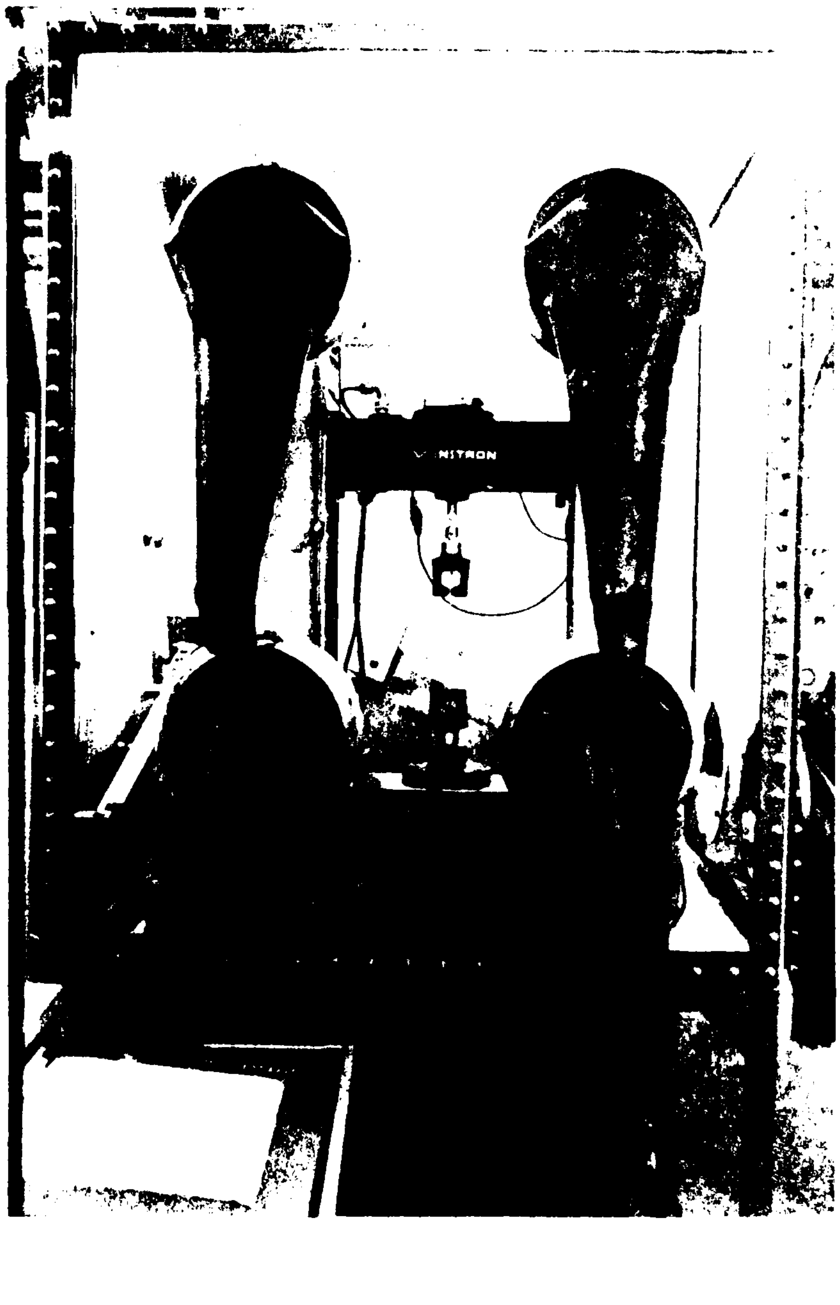

Figure 8. Close View of Box No. 4.

No.	Equipment	Main Purpose	Auxilary Purpose
1	5 Glove box system connected by antechambers	Preparation and charaterization of new sulphur, halide and chalcohalide glasses in a dry nitrogen atmosphere	
	Glove box #1	Melting new glasses and preparing glass fiber preform	Heat treatment and annealing of glass sample
	Glove box #2	Raw materials and sample preparation	
	Glove box #3	Optical properties and structure charaterization	
	Glove box #4	Mechanical properties measurement	
	Glove box #5	Horizontal preform fiber drawing and feasible for vertical fiber drawing set-up	
2	Analytical balance	Determination of sample weight	Weight loss measurement
3	Diamond saw	Adjustment of sample size	parallel surface production
4	Polisher/Grinder	Sample surface preparation	
5	Ultrasonic cleanser	Sample cleaning	removal of surface impurities and sono-gel preparation
6	Temperature controller w/ an associate unit	Control temperature of dual furnace horizontal preform fiber drawing equipment	
7	Programmable temperature controller	Control temperature of heat treatment furnace with different heating rate and dwelling time	

Table 1. Description of equipment and functions.

No.	Equipment	Main Purpose	Auxilary Purpose
8	SCR power source controller	Regulate power output to furnace	
9	Millivolt source	Calibration of thermocouple	Calibration of equipment
10	Infrared spectrophotometer	Measurement of IR transmittance of sample	Obtaining structural information
11	UV-VIS-NIR spectrophotometer	Measurement of UV-VIS-NIR transmittance of sample	Obtaining structural information
12	Mini-Instron	Tensile strength measurement of halide/chalcohalide/non-oxide glass fiber	Measurement of Modulus of rupture and compressive strength of bulk sample
13	Microscope w/ video attachment	Observation of fiber or bulk sample of halide, chalcohalide and non-oxide glasses prior to atmospheric exposure	Observation of crystallization, phase-separation and inhomogeneities of sample
14	Thermal Analyzer System		
	TGA (25°C - 1000°C)	Measurement of weight loss/gain versus tmperature	Organic burnoff, oxidation, carbonization and reaction kinetics of sample
	DTA (25°C - 1550°C)	Measurement of temperature difference between sample and standard versus temperature with controlled heating and cooling rate	Reaction kinetics and phase transformation kinetics and glass transition temperature determination
	DSC (25°C - 600°C)	Measurement of energy difference between sample and standard versus temperature with controlled heating and cooling rate	Heat capacity, reaction kinetics, phase transformation kinetics and glass transition temperature determination, and encapsulated sample observation

Table 1, Continued. Description of equipment and functions.

No.	Equipment	Manufacturer/Vendor	Cost	(Shipment cost)
1	1 Controlled Atmosphere four glove box system, dry train, oxygen analyzer and moisture analyzer.	Vacuum Atmosphere Co.	$ 76,356.00	(included)
2	1 Analytical balance Sartorius 1801 MP8	VWR Scientific	$ 1,275.00	(included)
3	1 Diamond saw w/ accessories Low speed Isomet	Max Erb Instrument Co.	$ 2,104.00	(included)
4	1 Polisher/Grinder w/ accessories Minimet	Max Erb Instrument Co.	$ 1,828.00	(included)
5	1 Ultrasonic sample cleanser Ultramet III	Max Erb Instrument Co.	$ 225.00	($ 4.08)
6	1 Temperature controller w/ an associate unit Model 810	Eurotherm Corp.	$ 1,010.00	
7	1 Programmable temperature controller Model 812	Eurotherm Corp.	$ 840.00	($ 37.75)
8	3 SCR power source controller Model 831	Eurotherm Corp.	$ 840.00	
9	1 Millivolt source Model 239	Eurotherm Corp.	$ 825.00	
10	1 Infrared spectrophotometer Model 1330	Perkin Elmer	$ 13,110.00	($ 107.96)
11	1 UV-VIS-NIR spectrophoto-meter Model 330	Perkin Elmer	$ 31,700.00	($ 134.59)
12	1 Mini-Instron w/ modification Model 1122	Instron Corp.	$ 28,960.00	($ 558.09)
13	1 Microscope w/ video attach-ment	Max Erb Instrument Co.	$ 8,860.80	(included)
14	1 DTA/DSC/TGA Thermal Analyzer system w/ Data station System 4	Perkin Elmer	$ 58,700.45	($ 285.54)
		Subtotal :	$226,634.25	($1,128.01)
		TOTAL :	$227,762.26	

Table 2. Equipment purchased.

Name of User	Equipment Number 1	2	3	4	5	6	7	8	9	10	11	12	13	14
Dr. Jas Sanghera (Postdoc)	x	x	x	x	x				x	x		x	x	
Dr. Rui Almeida (Professor)	x	x	x	x	x				x	x			x	
Dr. Hiroyuki Nasu (Postdoc)	x	x	x	x	x				x	x			x	
Sun-Youn Ryou (Ph.D. Student)	x	x	x	x	x	x		x	x	x		x	x	
Jong Heo (Ph.D. Student)	x	x	x	x	x	x		x	x	x			x	x
Dave Reinker (Master Student)	x	x	x	x	x								x	
Edward Pope (Ph.D. Student)							x	x		x	x			x
Azar Nazeri (Ph.D. Student)	x						x	x						x
Mike Borden (Master Student)														x
Mary Colby (Ph.D. Student)							x	x	x					x
Kuo Chun Chen (Ph.D. Student)							x	x	x	x	x			x
Ting Yuen (Ph.D. Student)					x		x	x	x	x	x			x
Deanne Yamato (Undergraduate)	x	x	x	x	x	x		x	x	x		x	x	
Rafael Zaldivar (Undergraduate)										x				x

Table 3. Members of the Ceramics Laboratory at UCLA using the new facility.

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